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(U) WASHINGTON UNIV SEATTLE DEPT OF CHEMISTRY
D J VELTKAMP ET AL. 01 NOV 84 TR-31 N00014-75-C-0536

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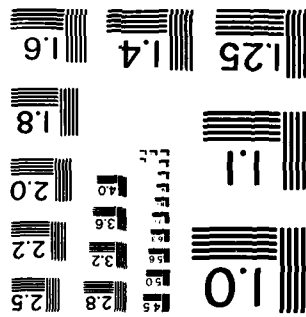
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Chemical Information from Ion Scattering Spectrometry

by

David J. Veltkamp and Bruce R. Kowalski

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in

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ABSTRACT



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Ion scattering spectroscopy (ISS) is a well established surface analysis technique yielding semi-quantitative elemental composition with sub-monolayer sensitivity. Poor sensitivity to low mass elements, limited resolution of high mass elements, and the lack of an ability to easily determine molecular species on the surface have limited the usefulness of ISS in surface analysis. Recent observations have shown oscillations in the scattered ion yield with incident energy for certain elements related to the neutralization probability of the ion-atom pair. Presented are oscillatory ion scattering spectroscopy (OISS) curves for a series of closely related lead compounds with the incident energy of the helium probe ion ranging from 500 eV to 4000 eV. These data show OISS to be very sensitive to the chemical environment of the target atom and so can be used to positively identify the chemical species in cases where normal ISS cannot. The use of OISS as a surface probe yielding chemical composition information greatly expands the usefulness of ISS while at the same time solving some of its inherent limitations. For those types of analyses requiring molecular information about the surface monolayer, such as catalysis and chemisorption studies, OISS may become one of the most powerful probes into this complex region.

Chemical Information from Ion Scattering Spectrometry

Introduction

In the simplest, or classical, form of ISS a beam of low energy (100 to 3000eV) noble gas ions bombards a surface and the energy of those ions undergoing single binary elastic collisions (SBEC) with the surface target atoms is measured. According to this simple SBEC (or billiard ball type collisions) model, the ratio of the scattered ion energy to the primary, or incident, ion energy (E/E_0) is related to the mass of the scattering surface atom as would be expected from classical physics. By scanning the spectrum of scattered ion energies one obtains peaks which correspond to the elemental mass composition of the sample surface. The yield of scattered ions, I_i , from a target atom in a sample is given by

$$I_i = I_0 N_i d\sigma_i/d\Omega \Omega P_i T D K \quad (1)$$

where I_0 is the primary ion current, N_i is the surface concentration of species i , Ω is the solid acceptance angle of the analyzer, T and D are the transmittance and sensitivity of the analyzer and detector, respectively, and K represents any other appropriate conversion factors. The remaining two terms in equation 1 are the ion survival probability, P_i , which includes both oscillating and non-oscillating ion neutralization pathways, and the differential scattering cross section, $d\sigma_i$; which varies with E_0 , the mass ratio (M_1/M_2), the scattering angle, and the

interaction potential. Since both $d\sigma_i$ and P_i are dependant on the incident ion energy, E_0 , any change in E_0 should result in a variation in the observed scattered ion intensity. As we will show later, the sensitivity of the ion yield to the incident ion energy can be used by the surface analyst to extend the capabilities of ISS and obtain additional information about the sample.

This relatively simple technique offers the advantage among surface analysis methods of very high surface selectivity, with a sensitivity of approximately 10^{-3} to 10^{-6} monolayer. Unfortunately, there are several problems in classical ISS which have limited its applicability. The mass resolution attainable by ISS is somewhat less than ideal particularly for the higher Z elements and is made even worse by the relatively broad peak widths and high backgrounds that are frequently found in ISS spectra. Also, since ISS gives only elemental information, identification of molecular species present on a surface is either difficult or impossible depending on the complexity of the sample.

Having emerged only in the mid-sixties, ISS is a very young field and recent developments indicate that it has great potential yet to be explored. Studies of multiple scattering events and shadowing have led to the ability to determine surface structure and orientation using ISS.^{1,2} Recently, the extension of ISS as an imaging technique has made it possible to obtain "chemical pictures" or "maps" of the spatially resolved chemical composition on a surface.³

Another promising development is the discovery of oscillatory ion scattering spectrometry (OISS) first noticed by Erickson and Smith for ion-solid collisions in 1975.⁴ They observed oscillations in the backscattered ion yield as the incident ion energy was varied for $^4\text{He}^+$

scattered from Pb, Bi, Ge, In, and InAs. A similar phenomenon was known to occur in the gas phase and was thought to be due to oscillations in the ion neutralization probability (P_i in equation 1) of the ion-atom interaction.^{5,6} A model to explain these oscillations was proposed based on a quasi-resonant charge exchange between the Helium ion's partially empty 1s orbital and the target atom's d orbital electrons. Resonant or quasi-resonant charge exchange requires that the ion-atom pair have either a pair of degenerate (resonant) or nearly degenerate (quasi-resonant) states. As the ion-atom pair approach each other, a time dependent potential owing to the interaction splits the formerly degenerate levels and the electron probability density oscillates between the two nuclei while they are in close proximity to each other. The final states at the end of the charge exchange process depend on the amount of time the system was in resonance and the energy level differences between the ion and the surface atom. This hypothesis is supported by the observation that only those elements having d electron energy levels within 10 eV of the first ionization energy of He (24.6 eV) exhibit the oscillatory structure.

The exact form of the OISS structure for a given element seems to vary with its physical and chemical state; and, as such, it provides a sensitive probe of the exact chemical environment of the element. As mentioned above, the OISS effect is thought to be due to a resonant or quasi-resonant charge exchange process and the evidence favoring this mechanism is substantial and has been well reviewed.^{7,8,9}

The actual form of the charge exchange mechanism is of course to be found in the quantum mechanical calculations of the interaction potentials and orbital overlap for the system of surface atoms and the incident ions under study. Although several researchers have attempted to model the OISS

curves based on quantum mechanical calculations, their results have not provided an adequate model for predicting the observed OISS spectra.¹⁰

In Figure 1 we have plotted the ISS ion yield intensities as a function of the incident ion energy for several of the elements in the region of the periodic table known to exhibit the oscillatory effect. One can clearly see the transition in the overall appearance of the curves as one traverses the rows of the periodic table. The general shape of the curves are due to the competing effects of the differential scattering cross section term, $d\sigma_i$, and the non-oscillatory charge exchange mechanisms in the ion survival probability term, P_i , from equation 1 above. Superimposed on this general curve shape are the oscillations due to the quasi-resonant components of P_i . The transition in the strength, or intensity, of these oscillations along the row of the periodic table correspond to the passing through a minimum in the energy difference between the d orbital energies of the sample atoms and the incident ions ionization energy. This leads to a maximum in the oscillations consistent with the quasi-resonant charge exchange mechanism explained above.

This paper will describe the instrumentation and experimental procedures used in this laboratory for the collection of OISS spectra. Experimental evidence will show the technique to be very sensitive to changes in the chemical environment of the target atom resulting in distinctly different OISS spectra for a range of similar compounds. The uniqueness of the OISS spectra can be used to positively identify chemical compounds, extending the abilities of conventional ISS. It is the sensitivity of OISS to the chemical state of the target atoms at the surface atomic monolayer that gives OISS the potential to become an

important new probe in surface analysis.

EXPERIMENTAL

All spectra were collected on an 3M (now Kratos) ISS/SIMS model 535-VX Instrument interfaced to a Digital Equipment Corporation LSI 11/23 microprocessor. The ion scattering spectrometer consists of a cylindrical mirror analyzer (CMA), an ion gun mounted co-axially within the CMA, and a channeltron electron multiplier (CEM) mounted in a stainless steel ultra high vacuum (UHV) chamber. This configuration produces an ion beam normal to the sample plane (although the sample may be tilted away from the normal) and a conical 138° solid acceptance angle to the CMA. Samples are mounted onto a stainless steel sample holder with up to five samples loaded at a time. Sample positioning relative to the ion beam and the sample orientation to the beam axis is accomplished using sample current imaging and a mechanical 3-D manipulator feedthrough into the UHV sample chamber. Software was developed to drive the computer interface allowing the ion beam energy, ion beam location on the sample, the counttime per channel, cylindrical mirror analyzer (CMA) pass voltages, and the ion counting detection via an A/D converter connected to the ratemeter output to be controlled by MACRO-11 subroutines called from a FORTRAN program. The collected data could then be transferred directly to a VAX 11/780 for further data processing and plotting.

In order to reduce the time required to obtain the OISS spectra and the large data storage requirement (1000 data points for each full ISS spectra at each incident ion energy), a link scanning approach was developed for the collection of the data. Instead of collecting the

entire ISS spectrum at each incident ion energy, the CMA analyzer plate voltages were restricted to sweep through a small window about the energy of the peak of interest and only that portion of the spectrum was recorded. Since the abscissa in ISS is calibrated as E/E_0 , changing the incident ion energy causes the position of the ISS peaks to shift to higher energy ratio values for increasing E_0 . Rather than recalibrating the energy ratio axis for each new incident energy, the data collection window was moved, or shifted, with each increment in the incident energy. The appropriate shift factor required was obtained by measuring the difference in the peak's location at two different incident ion energies and the ratio of the change in peak location to the change in incident energy was used to keep the data collection window centered on the moving ISS peak. In this way the micro-processor is able to set the incident ion energy, scan the energy window about the peak, record the scattered ion intensities, and repeat the process for each new increment in E_0 . Subsequently, the measured signal intensity along with the corresponding E_0 value is written to disk and later used to produce the OISS curves.

The vacuum chamber was pumped to a stable base pressure of 2×10^{-8} torr after the samples had been introduced and then backfilled to a pressure of $5-10 \times 10^{-6}$ torr with the probe gas. The probe ion used for generating most of the spectra was $^4\text{He}^+$ (99.95%) supplied by Monsanto Research Corp. All samples were either amorphous metals or AR grade powders pressed into pellets and cleaned by $^4\text{He}^+$ ion bombardment, at low ion current to minimize surface damage, reduction, or other ion induced artifacts, until the ISS spectra showed no variation with time. In the case of insulating samples, charge buildup was avoided by the standard technique of flooding the sample with thermal electrons. Typical data

collection parameters used to produce the OISS spectra are listed in Table 1.

RESULTS AND DISCUSSION

Erickson and Smith realized the utility of the OISS spectrum as a finger-print to resolve elements such as Bi and Pb which are unresolvable using conventional ISS. Moreover, their comparison of the In oscillatory ion yield curve from pure In versus that from an InAs crystal was the first indication that this phenomenon may provide useful analytical information about chemical bonding on the surface. Unfortunately, the technique of OISS has not found much acceptance or use in the field of analytical chemistry despite its potential for removing some of the inherent limitations in ISS. Part of this may be due to the lack of a complete understanding of the neutralization process which has limited the ability to predict the oscillatory curve or its correlation to the physical properties of a given compound.

Figure 2 shows the lead OISS spectra for several lead compounds collected in our laboratory. From the figures one can clearly see the effect of the chemical environment on the lead OISS signal, both in the general spectral envelope and in the position and intensity of the oscillations. The direct interpretation of these curves in terms of meaningful physical or chemical parameters (i.e. electronegativity, bond strength, coordination number, bond distance) has proven to be problematic and to date no such parameters have been identified. As a result of our work in attempting to model the OISS spectra for a series of lead compounds, we have seen that the OISS curves are composed of sums of

oscillating terms and cannot be modeled by a single simply oscillating term in P_i . This is further supported by the spectral power density function analysis of the OISS spectra performed by Christensen et. al.¹¹ which shows more than a single frequency component in the power spectrum for all the OISS spectra they studied. The use of principal component analysis, which has proven to be useful in determining the underlying factors which determine the spectra in other similar data analysis situations^{12,13,14}, in this case gave component axes which were not directly related to chemical information.

Even in the absence of a direct interpretation, however, the sensitivity of the OISS signal to the chemical environment can be used advantageously to extract information not available in conventional ISS. Since the nature of the oscillations in the OISS spectra depend on the chemical environment and the electronic energy levels of the target atoms, OISS provides an extended lever with which to distinguish elemental identity or chemical composition over that of conventional ISS which relies only on the atomic mass of the sample atoms. This can be seen by the OISS spectra for lead carbonate and lead acetate in Figure 2 which show very marked differences even though the elemental composition is very similar and the two compounds would give nearly identical ISS spectra. By developing an extensive library of OISS spectra for a wide range of compounds and using a library matching or fingerprint identification scheme it would be possible to unambiguously identify the chemical species of an element based on their OISS spectra.

Figure 3 shows the OISS spectra for Pb, PbO, PbO₂, and Pb₃O₄. It should be clear that positive identification among the oxides would not be possible using conventional ISS since each compound contains the same

elements and only the stoichiometry is different. Ratios of peak heights have been used in this situation to distinguish between such similar compounds with normal ISS but the method is approximate at best and requires the analyst to measure all possible stoichiometries under exactly identical conditions to be successful. Even then, the results are limited by the semi-qualitative nature of ISS. However, by using OISS spectra and a library of OISS standards, positive identification can be easily accomplished. It is the ability to distinguish chemical information on the basis of the oscillatory ISS pattern that gives this technique its potential to become an important tool in the analysis of surfaces.

Another area of interest in surface analysis is in the study of adsorption of gases on a substrate. Preliminary results indicate that OISS, with its very high surface selectivity, may prove to be a useful method for exploring this problem. Figure 4 shows the OISS spectra corresponding to lead metal with varying degrees of oxygen coverage. The spectra were obtained by exposing a lead surface to an oxygen environment (approx. 10^{-4} torr for two hours) and recording the OISS signal as a function of ion sputtering time. As can be seen the resultant curves show the strong influence of the oxygen coverage on the lead metal OISS signal. Principal component analysis (PCA) of the oxygen exposed lead OISS spectra verified the presence of a second component needed to describe the spectra while only one component is found for the OISS spectra for pure lead. Additional work is needed to determine if these observed changes in the OISS spectra are due to chemical interaction between the adsorbed gas and the substrate, physical effects such as shielding of the lead atoms by the oxygen covering, or the additional non-oscillatory neutralization of the probe ions by the adsorbed gas. The presence of the second component does

indicate however, that the introduction of oxygen onto the lead surface has altered the chemical environment around the lead atoms enough to change the OISS spectra. Unfortunately, our instrument has only a medium to high range vacuum (10^{-8} torr) and fixed scattering and acceptance angles which severely limit studies of adsorbed species. Also, the unavailability of some confirmatory technique such as LEEDS, precludes us from determining the binding sites, surface structure, or the actual surface coverage of the adsorbed gas. However, these preliminary results do indicate that, with some additional work under more favorable instrumental conditions, the technique of OISS may be able to play an important part in the study of physical and chemical adsorption of gases onto solid substrate surfaces.

There are currently two main limitations to the usefulness of OISS as an analytical tool. The first is the lack of a readily available parameter to relate the observed oscillatory spectra to the chemical state of the element. The lack of a "chemical shift value" for the oscillatory curves implies that in order for the technique to be used for a wide range of samples in a general way there must be a large enough spectral library of OISS curves to allow fingerprint matching of the sample spectra with a previously measured spectra. Such a library is currently being compiled and includes the OISS spectra of many previously unstudied compounds of which only a few are reported here.

The other main limitation to the usefulness of OISS is the limited number of elements known to exhibit the oscillatory behavior. Presently only nine elements have been shown to give oscillatory ISS curves. Fortunately, the elements currently available to this technique are of wide interest in several areas, including semiconductors, fabrication, and

catalysis. Recent work by Baun¹⁵ has shown that elements which do not exhibit the oscillatory effect alone can (by combining with certain other non-oscillatory elements) give OISS curves in specific compounds. This indicates that elements could be "shifted" into the oscillatory region and be studied by OISS through influences on their chemical environment. Another possibility for expanding the range of compounds which can be studied with this technique is to look at the energy dependent ion yield curves of those elements which do not exhibit oscillations. Since the yield patterns for these elements are still influenced by their chemical environment it is conceivable there is sufficient variation in these non-oscillatory yield curves with chemical identity to be analytically useful.

The sub-monolayer sensitivity of ISS makes the technique of OISS attractive for the study of catalysis, corrosion, adhesives, and chemical or physical adsorption. The addition of chemical composition information from OISS on the surface monolayer enhances the contribution ISS makes towards a more complete understanding of these complex processes.

The current state of OISS can be said to be limited by a quantum mechanical understanding of the neutralization process and the lack of the ability to accurately generate or predict OISS curves from theoretical calculations. Without this theoretical background the technique may be limited to the confirmatory types of analysis such as those outlined in this paper or those situations where the surface sensitivity of ISS and the improved chemical sensitivity of OISS can combine to give unique types of information on certain systems.

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CREDIT

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TABLE 1 TYPICAL DATA COLLECTION PARAMETERS

INSTRUMENT PARAMETERS

Base Chamber Pressure	2×10^{-8} Torr
Backfilled Pressure	$5-8 \times 10^{-6}$ Torr
Ion Beam Dia. at 1" from Target	450 μm (FWHM)
Ion Beam Current	500 nAmps
Electron Ionization Energy (E_e)	140 eV
Electron Emission Current (I_e)	15-20 mAmps
Ion Source Filament Current (I_{fil})	2.50 Amps (controlled with feedback to keep I_e constant)
Channel Electron Multiplier (CEM) Gain Voltage	2660 Volts

SCAN PARAMETERS

Starting Incident Ion Energy	500 eV
Final Incident Ion Energy	4000 eV
Ion Energy Step Size	10 eV
Scan Window Width	50 Channels
Counttime/Channel	100 micro-seconds
Channel Resolution	0.1% of Energy Ratio
Number of Co-added Scans	3

Figure #1. Energy dependant ion yield curves for the series of elements near the oscillatory region of the periodic table (not shown are spectra for Au, Tl, Pb, and Bi which also followed the same general trend in appearance as the two rows of the periodic table shown above). All OISS spectra are normalized for ion current and detector transmissivity.

Figure #2. OISS spectra for a series of four related lead compounds and three lead halides. Spectra show the inherent sensitivity of OISS to the chemical environment of the lead atoms.

Figure #3. OISS spectra for lead metal and three common lead oxides.

Figure #4. Series of spectra showing the ion yield (in arbitrary units) versus incident ion energy for pure lead foil with varying amounts of oxygen coverage. Note that the pure lead signal is higher than the signal from the exposed lead at incident energies below approximately 1600 eV while above this value the relative intensities of the curves are reversed.

Fig. 1

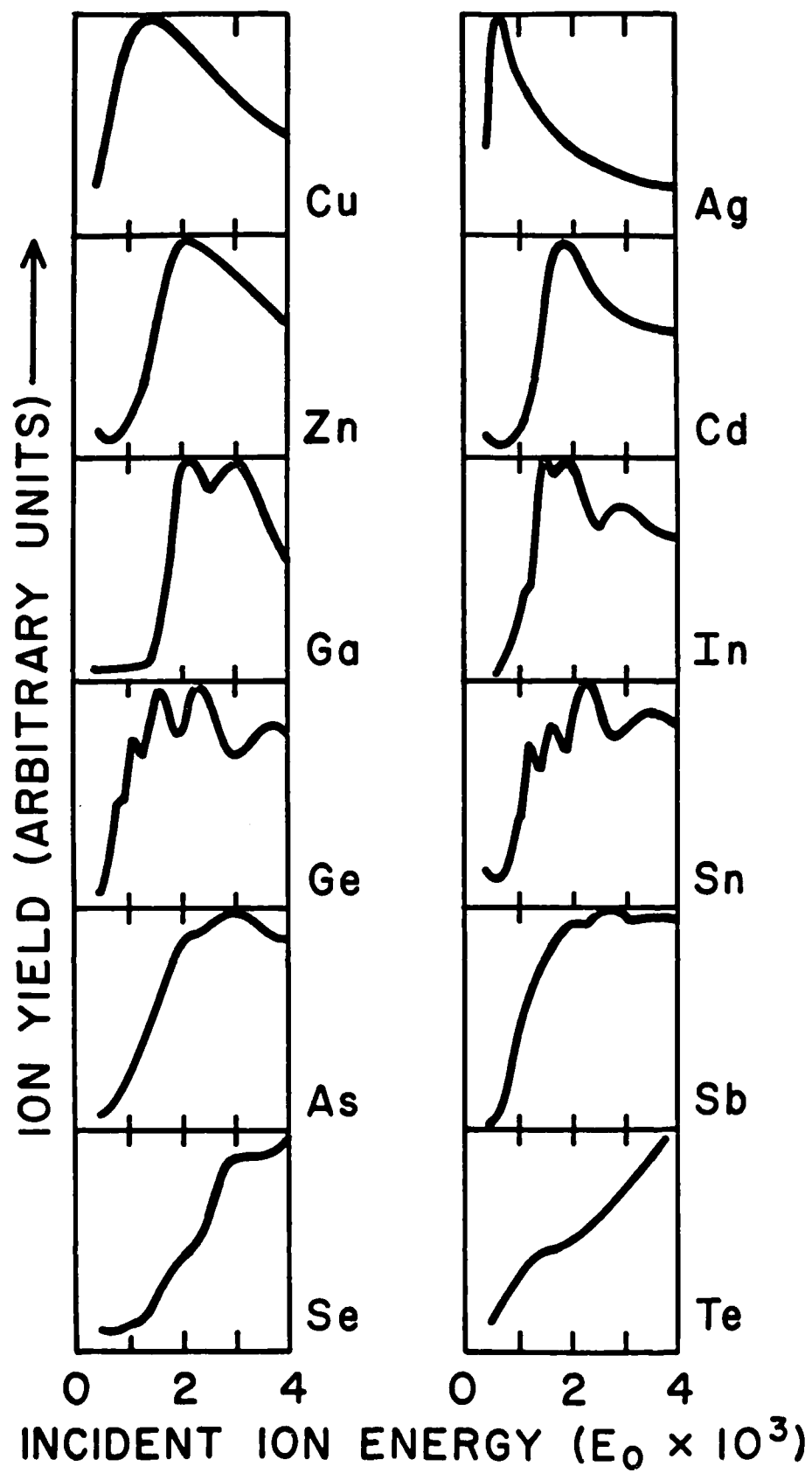


Fig. 2

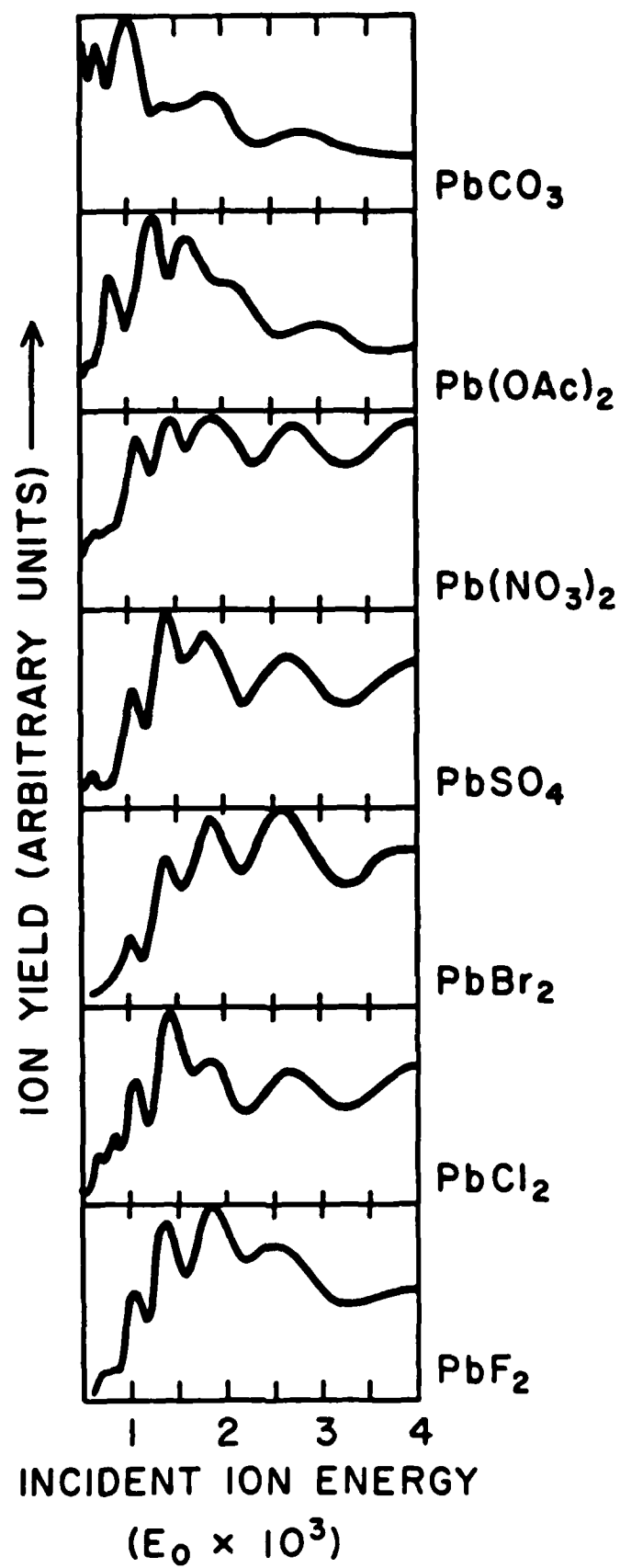


Fig. 3

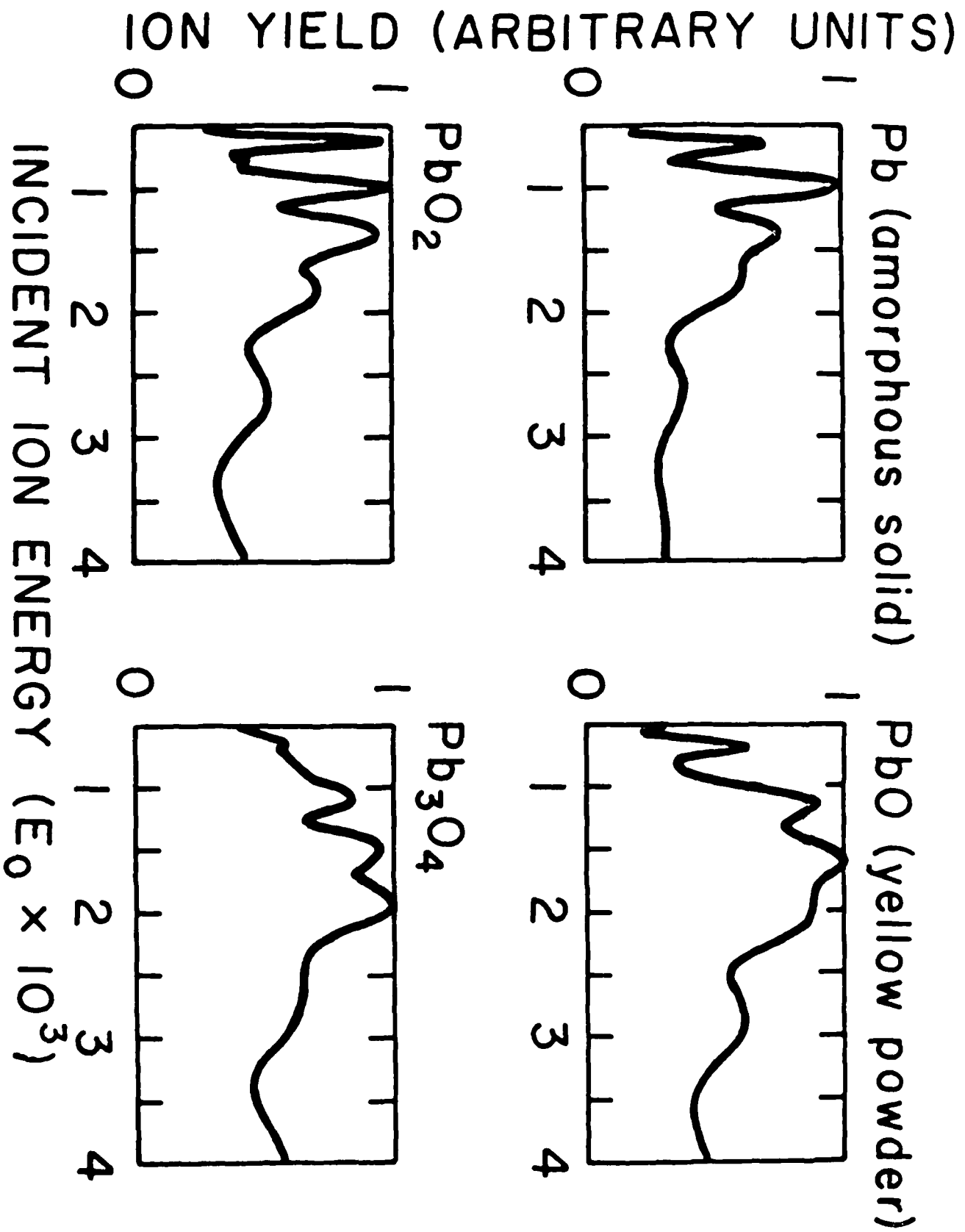
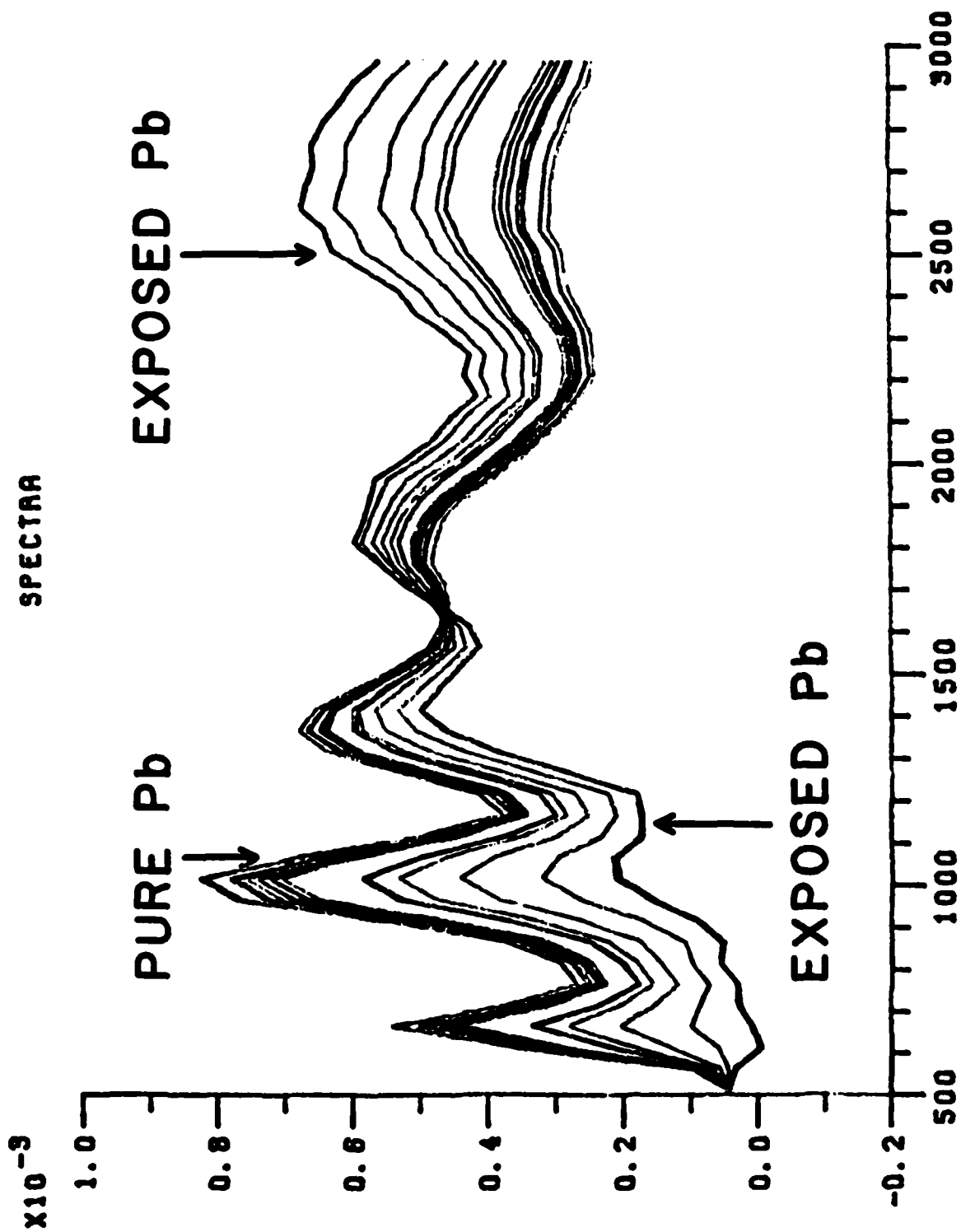


Fig. 4



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